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sidelights on SAND and SILICON

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DIRECTORATE OF CHEMICAL SCIENCES (AFOSR)

Silicon, the second most abundant element on earth, is familiar to most of us as an ingredient of sand and also of silicones which are used as polymers in high temperature hydraulic fluids and rubbers. More recently silicon has been in the limelight in the field of semiconductor devices and solar power cells for satellites. In spite of the technical knowledge of silicon and its compounds, there is still much completely baffling and unknown about the fundamental basic chemistry of bonding in silicon compounds in general.

To help unravel some of the complexities of this most important element, Dr. West at the University of Wisconsin, under the sponsorship of the Chemistry Directorate, AFOSR, is conducting a research program on the basic chemistry of silicon. Some of his contributions

in the short period of four years are as follows:

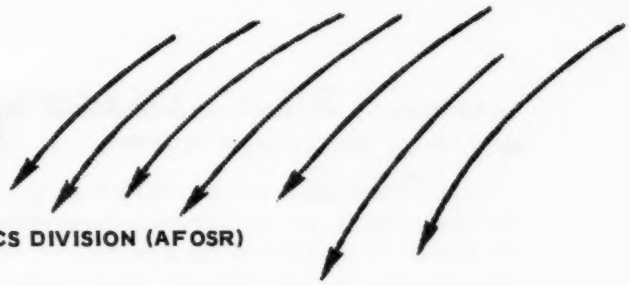
New Aromatic Anions - New four and five ring systems known as the croconate and rhodizonate ions have been shown to have the same type of Pi bond resonance first found in benzene and then extended to other carbon ring systems including "squaric" acid and also to mixed ring systems, for example, borazole and boroxole. Cyclic resonant structures are of interest both in the study of semi-conductors and in the very recent discovery of vitreous organic "lasers".

Nature of the Silicon Oxygen Bond - New knowledge on the basic chemistry of the Pi bonding of the p electrons of oxygen to fill the vacant 3d orbitals of silicon. In this live field, West and his students contributed a number of significant papers particularly dealing with the infrared shift of the Si-O bond and its relation to intramolecular association of silanols. It was shown that this bond was decidedly stronger than in alcohols. Incidentally, the Russians have also been very active in the field of Raman and infrared spectra, particularly in the basic chemistry involved in the spectral shifts due to such factors as association.

Additional Silanes of Olefines - The work on the addition of silanes or silicon compounds having one or more attached hydrogen groups to olefines is related to the currently booming field of "hydroboration " a new method of organic synthesis of alcohols. It has resulted in three papers on the mechanism of the ionic addition of trichlorosilane to methycyclohexene.

Silicon-Nitrogen Dative Bonds - The chemistry of silicon-nitrogen dative bonded compounds is a new vein of knowledge explored by West. It is closely related to similar extensive studies in boron-nitrogen compounds. An incidental observation of West was that triphenyl-silyl azide was surprisingly an extraordinary stable material. West assumes the explanation to be in the strong pi bonding between nitrogen and silicon which causes silyl azides to have a linear structure in contrast to the ordinary organic azides which have a bent structure. What the explosives chemists will make of this new information is anybody's guess. Fundamental knowledge in chemistry pays off in knowledge in many fields of direct interest to the Air Force. This work seems likely to do so quickly in more than one area of practical interest.

MAGNETIC FIELDS FROM SPACE



RAY R. HEER, JR., NUCLEAR PHYSICS DIVISION (AFOSR)

A general disturbance in the magnetic field of the earth generally follows solar flares by from 1 to 3 days. The disturbances are probably caused by the interaction of particles from the sun (and the magnetic fields which accompany them) with the earth's magnetic field. The time delay between electro magnetic detection of the solar flare and the geomagnetic disturbance gives us a clue about the plasma (the magnetic cloud may be thought of as a plasma) speed from sun to earth. It sometimes happens that these distu rbances are accompanied by what is called a Forbush decrease in the normal flux of the cosmic radiation. This Forbush decrease is a rapid decrease in intensity followed by a gradual recovery to pre-storm level, the amount of decrease varying with type of radiation, being as high as 30% for the nucleonic component and as low as a fraction of a percent for the very energetic meson component. This energy dependence is one of the things which indicates the presence of an extraterrestrial magnetic field near the earth. Air Force capability in space is intimately related to understanding and, hopefully, learning to obviate unwanted complications caused by these fields.

An AFOSR grantee, Professor Ismael Escobar V. of La Paz Bolivia has been studying Forbush decreases among other parameters of the cosmic radiation, in an attempt to advance the understanding of the magnetic fields. Specifically, he is seeking to identify the nature of low energy solar emissions and the magnetic fields which contain them.

The La Paz group is located very near the geomagnetic equator. They, along with researchers at other latitudes, have been able to detect small preonset, or precursory, increases of cosmic ray intensity before a Forbush decrease and to conclude that this preonset increase is latitude dependent. The preonset increase may be compared to a ship's bow wave; the cosmic rays which occupy the space between sun and earth being pushed toward earth by the magnetic cloud which is travelling from sun to earth. One would expect the lower energy particles to be more affected by such a mechanism and, indeed, it is at higher latitude stations, where the lower energy particles can penetrate the earth's magnetic field, that the largest preonset increase is

detected. Increases in equatorial latitudes appear to be less marked and to be longitude dependent.

The equipment at LaPaz is such that the flux, and its variations, can be measured in various directions. As is not surprising -- the directional instruments are called telescopes. There are three telescopes; one points toward the zenith and the other two point, respectively, 45^{o} E and W of zenith. The pattern of variations is quite similar in all of them but it has been noted that the W pointing telescope shows the largest decrease. This too is an indicator of greatest change in the low energy part of the spectrum since we know, from other experiments, that the lowest energy arriving primaries arrive preferentially from the West.

These studies of Forbush decreases also shed some light on the question "Is the primary cosmic radiation isotropic or anistropic?", i.e., are there source regions? This follows from the reasoning that complete isotropy would result in the same Forbush pattern for stations at the same latitude and at different longitudes. However, if there is anisotropy it should show up as a function of time at stations at various longitudes. The results are that stations at middle latitudes show the behavior expected for isotropy while equatorial stations show the opposite. It thus appears that low energy primary cosmic radiation is isotropic but there is some argument for anisotropy for high energy particles.

The speed of onset and recovery of a Forbush decrease is used to learn something about the shape of the solar gas cloud which gives rise to the decrease. It is necessary to use single events since the analysis becomes quite difficult when multiple events, i.e., decreases which overlap in time, occur. For single magnetic storms and the accompanying Forbush decrease the following conclusions which are consistent with all known parameters have been reached: A strong magnetic storm will generally be associated with a central solar meridian outbreak and both the decrease and recovery will be sharp. The recovery time will be about three days. A weak storm is associated with an outbreak which is far from central meridian and which may have a recovery period of 10 days. The strong storms are most probably confined to a narrow cone whereas the weak storm fills a considerable volume of space. As the storm decreases in intensity the magnetic fields in the solar plasma (and there is no doubt about the existence of the fields -- or there would be no decrease) break up into turbulent eddies and eventually become parts of the interplanetary magnetic fields.

COORDINATION COMPLEXES and CANCER CONTROL

BRANDES H. SMITH, LT., USAF, DIRECTORATE OF CHEMICAL SCIENCES (AFOSR)

To the scientifically cognizant person the terms dentate, ligand complex and chromatography usually conjure up impressions of techniques for separation of mixtures and purification of materials. The major effort in these areas has indeed been along such pragmatic lines. However, some recent work by Dr. Stanley Kirschner on metal complexes as related to cancer has stirred considerable interest. This interest is heightened when one speculates on the implications of some other results on multidentate ligand coordination complexes, gathered by Dr. Dale Margerum of Purdue University, under AFOSR support.

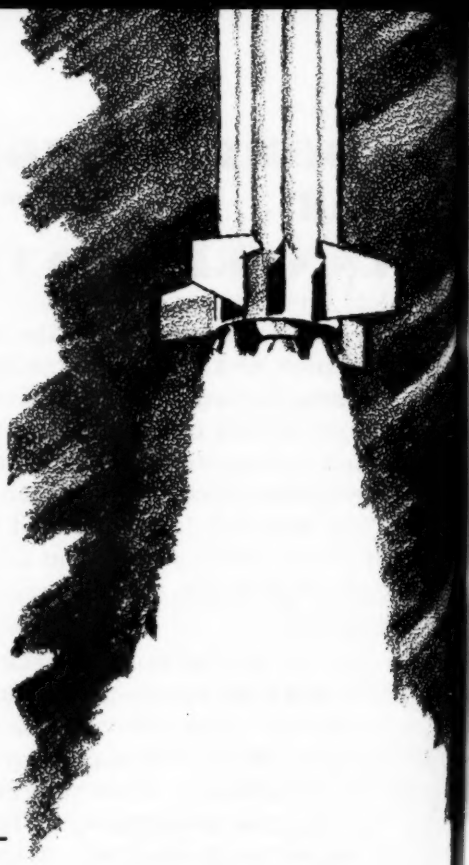
In one form or another, depending on the species, metals may be either carcinogenic or anti-carcinogenic in nature. It was found, in part by Kirschner and in part by others, that anti-carcinogenic metals, when complexed by organic compounds, may be used to treat some cancers. Also carcinogenic metals may be complexed, thus fighting the malignancy. These modes of operation have thus far been independently applied. Margerum's work offers hope for a simultaneous, double-barrelled attack and in a varied, yet highly selective, fashion.

In brief, he has discovered a coordination chain reaction mechanism which may allow for an organometallic complex selectively to introduce the cancer-fighting metal and immediately thereafter complex and thus sequester the cancer-causing metal. All of this may occur in a non-reversible fashion! This possible approach has its basis in several phenomena observed in Margerum's metal-complex work. Sheer bulk of the metal plays some part in assuring non-reversibility. In other cases two relatively inert complexes exert a synergistic effect. Though independently inert to start, when placed together they readily react and then, after exchanging metals, lapse back to inertness. There are even cases of two very similar metals exhibiting dissimilar behavior, one forming a strong complex while the other does not complex at all.

The work in this area has hardly begun. At its inception, no thought was given to the medical value; it was strictly a materials research program. But here again scientific results show their capability of cutting across disciplinary lines: basic chemical research providing another possible tool for the medical researcher.

A HIGH TEMPERATURE MEASUREMENT TECHNIQUE

THIOPHILOS ANDRADA, CAPTAIN, USAF
THERMOMECHANICS RESEARCH BRANCH
(ARL)



Temperatures in the range above 5000° F are presently being generated in rockets, arc heated plasmas, and advanced types of propulsion systems. Knowledge of these temperatures is necessary for complete evaluation of system performance, but are extremely difficult to measure. The problem is usually magnified by large variations throughout a given stream; for example, both radial and axial temperature gradients are encountered.

As a result of an unsolicited proposal, the Aeronautical Research Laboratories have been sponsoring a research investigation under the direction of Dr. W. H. Giedt at the Advanced Technology Laboratories, Mountain View, California, that promises a new and useful method for accurately determining the gas temperature in high temperature plasmas. The approach involves, in essence, the use of two thermal elements (thermocouples, thermistors, etc.) having identical dimensions but unequal thermal capacities. If successful, the experimental apparatus that is being developed will be utilized in the plasma arc facility of the Thermomechanics Research Laboratory of the ARL. A comparison of this method will be made simultaneously with other high temperature techniques (spectroscopic and infrared) on a long (approx. 36-inch) arc discharge capable of generating temperatures up to 20,000° F.

No temperature sensor presently available can be continuously immersed in a gas stream or plasma above 5000° F without rapidly melting. The approach suggested by Dr. Giedt to overcome this limitation is to immerse the sensors in the high temperature stream for a very short time duration and record the temperature -- time

history of each sensor as shown in Figure I.

As previously mentioned, the thermal capacities of each element are not equal, hence one would obtain two different response curves as illustrated in Figure I. Thus, if one writes the heat balance equation for a sensor element, one obtains the following:

$$hA\ (t_g - t_s) = WC\ \frac{dT}{d\tau}$$

where h is the overall heat transfer coefficient, A is the area exposed to heating for each sensor, t_g and t_s are the gas stream temperature and the sensor temperature respectively, WC is the heat capacity of each sensor, and dT/d is the time rate of temperature change of each sensor, or the slope at any instant of time of the response curve of Figure I.

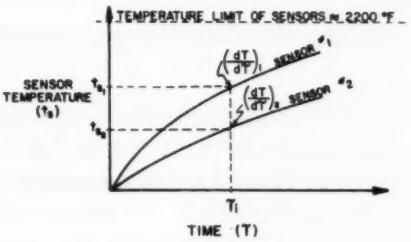


FIGURE-1

The technique is based on the assumption that if the geometry and dimensions of both sensors are identical, then the overall heat transfer coefficients of each sensor are equal, provided, the sensors are located in the same position in the stream.

The heat transfer coefficient, h, on each sensor is unknown; it depends on the type of gas, its temperature, pressure and velocity. However, if geometry and dimensions of each sensor are identical, the heat transfer coefficients on both sensors are identical. Therefore, since the overall heat transfer coefficient, h, and the exposed area, A , are equal for each sensor, one can then write the heat balance equation for each sensor, eliminate the product hA, and solve for the gas stream temperature, t_g. One then obtains a relation which states that the gas temperature is a function of the heat capacities of both sensors, which is known, the simultaneously measured instantaneous temperature and gradients of both sensors, but independent of the heat transfer coefficients which are not known. One then arrives with the following relation:

$$t_g = f\ (t_{s1},\ t_{s2},\ WC_1,\ WC_2,\ (\frac{dT}{d\tau})_1,\ (\frac{dT}{d\tau})2\ )$$

where subscripts 1 and 2 refer to the respective sensors. The heat

capacities (WC_1, WC_2) are constant and calculated for each sensor.

From the time-temperature record (Figure I), at any time τ one can evaluate the two sensor temperatures (t_{s1}, t_{s2}) along with their respective slopes,

$$\left(\left(\frac{dT}{d\tau_1} \right), \left(\frac{dT}{d\tau_2} \right) \right)$$

and calculate the gas stream temperature t_g.

The initial work of the contractor has emphasized an error analysis to find the conditions for selecting the proper ratio of heat capacities of the sensors, geometry of the sensors, and also the type of sensors.

The technique has been experimentally verified by using two cylindrical thermocouples of the same material and size but differing in construction (one was solid and the other hollow) to obtain a difference in heat capacities. The sensors were exposed to an oxy-acetylene flame and the first results were within 5 per cent agreement of standard measuring techniques.

The technique offers some excellent features when compared to spectroscopic techniques in that the evaluation of the results can be obtained very quickly; also, the simplicity of the system does not require a highly trained operator. Some of the disadvantages are the disturbance of the stream, due to the immersion of the sensors, and electrical noise effects when used directly in an arc discharge. Also, the accuracy of the results is dependent to a great extent on a precise evaluation of the slope of the temperature-time curves which would require a very sophisticated electronic differentiator system.

An analysis of a suitable read-out system along with further experimental tests are now being performed to establish the limitations of the technique with respect to its proposed uses in high temperature research.

definition of "FREQUENCY"

IONOSPHERIC PHYSICS LABORATORY (AFCRL)

A new technical term usually enters the language with a precise meaning. (It can mean exactly what the originator wishes it to mean, nothing more, nothing less.) As the word enters general usage, however, precision is often lost. The word is made to carry a larger and larger burden as shadings of meaning broaden. The user assumes that his definition is generally shared -- which isn't always the case.

A basic technical term is "frequency." To be meaningful, this term must always have an adjective, either implicit or explicit -- i.e., carrier frequency, modulating frequency, gyro frequency, plasma frequency, and so on. The lack of preciseness of "frequency" has several implications. These implications are discussed in a paper recently published in the Proceedings of the IRE by Dr. Kurt Toman of AFCRL. Dr. Toman's paper is quoted in part below:

"There exist two basic methods of measuring frequencies of electro- magnetic waves. One method uses resonance or counting circuits as in radio engineering. The other method employs prisms or gratings as in optics. At the basis of these measurements lie certain concepts regarding waves, like period, wavelength, frequency, etc. An abstraction to a periodic wave in its simplest form is a sinusoid of some frequency. A variety of more complicated waveforms can be synthesized by means of sinusoidal components of different frequencies using Fourier representations.

"For a sinusoid (waveform A) successive cycles are, by definition, identical. If the waveform in question is not sinusoidal, but is identically repeated (waveform B), it is again possible to attribute to this process a frequency. Since in both cases the waveform consists of identical cycles, we could define a frequency as 'the number of identical cycles per second' (definition I). The frequency of B is of the nature of a repetition-frequency, while the frequencies of its sinusoidal components are known as the frequencies of the harmonics.

"If a waveform C depicts a cyclic nature but the cycles are

not identical because of amplitude, phase, or frequency variations (e.g., radio emissions from artificial earth satellites, alpha-rhythm of brain waves, etc.), it is yet desirable to assign frequencies to these waveforms. If the definition frequency=number of cycles per second (definition II) is used with waveforms B and C, the latter is not distinguished from the former. With definition I a distinction between B and C is conceptually possible, since B can now be designated with a "frequency," while C falls for "average-frequency". If definition II is used with waveform C the cycles are only implicitly identical. With definition I the cycles of C are explicitly different. Here, the use of the connotation 'average-frequency' emphasizes the fact that the cycles are made to be identical, while the cycles of B are explicitly identical.

"It should be remembered that the word frequency originates from 'frequentia' (Lat: numerous, attendance, multitude) and was, of course, used before Maxwell's (1864) and Hertz's (1889) discoveries of radio waves. In the word multitude (relative frequency in statistics), for example, the identical nature of the entities whose multitude we seek is only, but indeed, implied. To determine the multitude of 2 dogs + 3 cats = 5 mammals, a new dimensional quality is introduced. From the point of view of being mammals, cats are identical to dogs. Thus, multitude reasonably interpreted implies, but does not explicitly refer to, multiple identities.

"In scientific disciplines one cannot be satisfied with implications. Because scientific knowledge grows through the interplay between theory and experiment, and since theoretical concepts always contain idealizing assumptions which must come to light before the theory is related to the experiment, definition I may be a useful conceptual tool for the interplay between theory and measurement. Instantaneous frequency, however, remains predominantly a mathematical tool, inaccessible to measurement but useful for deductions."

A NEW TECHNIQUE FOR RECORDING HEAVY PRIMARY COSMIC RADIATION AND NUCLEAR EVENTS

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The difficulties in the detection of primary cosmic radiation by photographic emulsions have been responsible for a continuing interest in the development of alternate detection devices. A new technique

developed by Professor Lawrence Slifkin at the University of North Carolina has been successful in initial tests for detecting both primary cosmic ray particles and high energy nuclear events. Professor Slifkin is doing this work under the sponsorship of AFOSR.

The technique is somewhat unusual in that it was originally devised to study radiation damage and imperfections in crystals, primarily dislocations. In initial dislocation studies, a silver crystal was bombarded by nuclear radiation. The energy lost by particles traversing the crystal produces a sudden temperature increase in a cylindrical region centered on the particle track. The excell thermal energy is rapidly dissipated, leaving a region with a high concentration of crystal imperfections such as dislocations. This track may be developed as follows. One surface of the crystal is exposed to pulses of ultraviolet light. A synchronously pulsed electric field sweeps the resulting photoelectrons through the crystal. Some of these photoelectrons become trapped at the imperfections in the track of the particles. Interstitial silver ions migrate to the trapped electrons and thereby form silver atoms; with successive pulses, the path of the cosmic ray particle or dislocation becomes delineated with silver. A pulse repetition rate of 1000/sec, for one to two hours is usually sufficient to delineate the dislocations.

An actual test of this procedure as a particle detector was recently performed with excellent results. Several crystals were packed in individual holders and the position of each was marked on an accompanying photographic emulsion. The crystals were flown at 123,000 feet for 8 hours and developed, upon return, as described above.

A comparison of the emulsions and the crystals demonstrated that all of the high energy particles of the C-N-O group and above produced developable tracks in the crystals. This new technique for detecting primary cosmic radiation and nuclear events has as its main advantages the absence of track distortion, the large thickness of crystals which may be employed, and the insensitivity to the low-Z component of cosmic radiation. This, as Professor Slifkin suggests, may make these crystals particularly useful for extended exposures in the study of the heavy component of cosmic radiation. Professor Slifkin reported this work at the 8th Scintillation and Semiconductor Symposium, held in Washington, D.C. on 3 March 1962. A more comprehensive report will be prepared and submitted to the Physical Review.

SILICON IS SCHIZOPHRENIC

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Berzelius opened a Pandora's Box when he discovered silicon in 1827. From that time there has been an expanding effort to explain and categorize the complex chemical behavior of this element. It has been referred to as a "metalloid" because it exhibits some of the properties of both metals and non-metals. Today, nearly everyone thinks of elemental silicon as a "semi-conductor" with the emphasis placed on its unique behavior in electronic circuitry. However, the technology of silicon compounds as applied to the ceramic arts is as old as mankind. Processes connected with the fabrication of pottery products and ceramic ware using alumina silicate (clay), feldspar (alkali-alumina-silicate) and flint (silica) as raw materials have been studied by more people and over a longer period of time than any other single chemical reaction. Even so, there are still many unsolved problems. Many paradoxical and controversial observations remain to be clarified.

In the search for new information which might lead to the development of improved high temperature materials, the Aeronautical Research Laboratories began experimental work more than 10 years ago on the binary system silicon-oxygen. The first results, "Silicon-Based Cermets and Related Observations" were presented at the 1953 Annual meeting of the American Ceramic Society. At that time silicon seemed to be especially favorable as a cermet component for applications in turbine buckets because of its low specific gravity of 2.33, a favorably low thermal expansion coefficient and a melting point of 2590°F. It was concluded that silicon could become an important engineering material if it would be possible to combine it with ceramic materials creating a cermet with similar favorable properties. The one aim, to develop a thermal shock resistant highly refractory cermet, was accomplished as numerous tests under simulated service conditions proved. Unfortunately, the final goal to find a cermet material with high impact strength could not be reached. In this respect, our experimental materials share the fate of all cermets which were considered hopeful, but finally were abandoned because the inherent brittleness of ceramics or cermets has not been overcome.

Many observations were made and recorded during this early work. However, the results could not be fully evaluated until recently when improved X-ray capability and more reliable methods of analysis were made available. The experimental "observations on the sideline" have been re-

studied recently by different methods. The results were presented as a paper entitled "ON FIBROUS SILICA, SILICON SESQUIOXIDE AND A SILICON-OXYGEN COMPOUND SIMILAR TO MULLITE" at the Meeting of the Basic Science Division of the American Ceramic Society in Schenectady (G.E. Research Laboratory) in October 1961.

The experimental conditions leading to the formation of a fibrous silica with amorphous structure had already been briefly discussed in the 1953 paper. The results now were interpreted in detail and brought in line with data published in 1953 and 1954 by a research team from the Technical University of Darmstadt, Germany, under the direction of Professor Dr. Ulrich Hofmann. After exchanging reprints, Professor Hofmann (now Director of the Chemical Institute of the University of Heidelberg) who is an old friend of the writer's, confirmed that our results were in complete agreement with his own. He also found that his silica fibers were amorphous.

Later on, two brothers Alarich and Armin Weiss at the Darmstadt Institute in 1954 published a paper "On the Fibrous Silicon Dioxide Modification" wherein they announced a new crystalline silica form with fiber structure and chain molecules. An important property of the new crystal modification is its short life when it comes in contact with traces of water vapor. This fact is certainly the reason that it was not discovered earlier.

Our own experiments have shown us that well developed fibers were obtained when silicon powder was fired in a furnace heated with natural gas where traces of sulfur are always present. It is hypothesized that the presence of sulfur favors the formation of fibrous silica since long and well-developed fibers of silicon disulfide crystals are known to exist. In the presence of water they change to an amorphous structure.

Our investigation of the suboxides in the system silicon-oxygen was considered a valuable contribution to a controversial subject by the Schenectady audience. In 1953 we had critically reviewed the literature known at that time. Concerning silicon monoxide, the black resin-like condensate (obtained by reacting molecular proportions of silica and silicon in vacuo) was believed to be a chemical entity. It was, e.g., completely soluble in HF and by X-rays exhibited one single broad ring with a d-spacing of 3.6 Å. In contrast, the fluffy brown condensate prepared in helium atmosphere indicated the presence of a diffuse ring of amorphous silica and clear patterns of elemental

silicon, providing that the suboxide has decomposed into SiO_2 and Si. In conformity with this observation the tan colored material was 68% soluble in HF leaving a residue of silicon. From determinations of weight gain when heated in air, the helium atmosphere product was found to fit generally the composition SiO. But we observed that the product was not uniform in color. Samples of lighter color had weight gains corresponding to the formulae Si_2O_3 and Si_3O_4, respectively.

Numerous publications have been critically reviewed since 1953. Some of these supported, while others seemed to disprove the existence of a true compound silicon monoxide. We expressed the opinion that over-emphasis should not be placed on the existence or non-existence of the silicon suboxides, but rather on the conditions controlling their stability. What is said for SiO is equally valid for Si_2O_3.

At the suggestion of Dr. L. Spialter of the Chemistry Research Laboratory of the ARL, a different approach was used for the preparation of silicon sesquioxide. The method consists of hydrolyzing silicon hexachlorodisilane (Si_2Cl_6) with subsequent heat treatment. Experimenting with this Si_2O_3 under various firing and atmospheric conditions and checking the results with X-rays and the petrographic microscope we have obtained data which throws light on the formation and stability conditions of this unusual compound.

In the last part of our recent paper we reported an unexpected reaction product obtained by heating silicon powder in a wet hydrogen atmosphere at 1760°C. After the firing, the silicon powder was completely transformed into a network of beautiful colorless crystals. A careful chemical analysis by Dr. R. W. Moshier, Chemistry Research Laboratory, revealed the surprising fact that it was not attacked by HF and that it contained 43.5% Al_2O_3. The result indicates that a reaction with the alumina support took place during the firing. The chemical composition of our reaction product fits closely to the formula $Al_2O_3.2\,SiO_2$, a compound normally called meta-kaolin. According to earlier investigations, meta-kaolin (resulting when Kaolin ($Al_2O_3.2SiO_2.2H_2O$) loses its water of crystallization on firing) gives no distinct X-ray pattern, but our reaction product did. A comparison with known X-ray patterns shows that it resembles α-Al_2O_3 very closely. But such an interpretation would be in conflict with: 1) the result of Dr. Moshier's analysis, 2) the well-known fact that alumina and silica react readily at temperatures above 1300°C.

Now it appears we have discovered a new compound in the complex

SiO_2-Al_2O_3 system. Additional work is underway to establish the true crystal structure.

In summarizing the results of our research effort, the statement can be made that we have contributed to our knowledge of the complex phase relations in the system silicon-oxygen. This is important in order to completely understand the chemical reactions occurring when silicon compounds are subject to high temperatures in various atmospheres. The Air Force has a direct interest in this type of basic information because silicides and other silicon compositions are among the structural materials and coatings developed to protect refractory metals against the corrosive attack of oxidizing atmospheres.

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